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Atomic Oxygen Durability of Solar Concentrator Materials for Space Station Freedom

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ATOMIC OXYGEN DURABILITY OF SOLAR CONCENTRATOR MATERIALS FOR SPACE STATION FREEDOM

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ABSTRACT

This paper reviews the findings of atomic oxygen exposure testing of candidate solar concentrator materials containing SiO_2 and Al_2O_3 protective coatings for use on Space Station Freedom solar dynamic power modules. Both continuous and iterative atomic oxygen exposure tests were conducted. Iterative air plasma ashing resulted in larger specular reflectance decreases and solar absorptance increases than continuous ashing to the same fluence, and appears to provide a more severe environment than the continuous atomic oxygen exposure that would occur in the low Earth orbit environment. First generation concentrator fabrication techniques produced surface defects including scratches, macroscopic bumps, dendritic regions, porosity, haziness and pin hole defects. Several of these defects appear to be preferential sites for atomic oxygen attack leading to erosive undercutting. Extensive undercutting and flaking of reflective and protective coatings were found to be promoted through an undercutting-tearing propagation process. Atomic oxygen erosion processes and effects on optical performance will be presented.

INTRODUCTION

In low Earth orbit (LEO), where Space Station Freedom (SSF) will operate, a harsh environment exists which can cause considerable damage to vulnerable materials. The environment includes atomic oxygen, UV radiation, thermal cycling, and micrometeoroid and debris bombardment[1]. The importance of ground based durability testing has become increasingly recognized as a necessity for evaluating the durability of materials proposed for SSF.

Atomic oxygen, the predominant species in LEO[2], is extremely reactive, particularly at ram impact energies ($\approx$ 4.5 eV). Therefore, materials susceptible to oxidation need to be protected when used in LEO. If pin hole defects exist in protective coatings on organic substrates such as graphite epoxy or Kapton[R], atomic oxygen erosion can occur due primarily to secondary impacts (scattered atomic oxygen impacts) where the substrate material is eroded away with the diameter of the eroded substrate area being larger than the diameter of the original defect. Erosion damage of this type is called atomic oxygen undercutting. Undercutting can result in the degradation of several materials properties. It may cause further damage to the protective coatings leading to optical performance degradation and potential structural failure. Atomic oxygen undercutting and its damaging effects have been studied extensively[3,4,5,6,7,8,9].

Electrical power for SSF may be increased during a growth phase by the addition of 25 kW solar dynamic power modules (SDPM)[10]. In a SDPM, a solar concentrator reflects and focuses solar

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radiation into the receiver of a heat engine. The efficiency of a SDPM depends on the ability of the solar concentrator to accurately direct the maximum amount of the sun's incident solar radiation into the heat receiver. It is critical that the concentrator has and maintains a high solar specular reflectance. The concentrator should also maintain a low solar absorptance during operation in LEO so that the concentrator materials do not become excessively heated. Overheating may lead to concentrator warpage and substrate damage.

First generation solar concentrator coupons under study are a sandwich type structure made of two sheets of graphite epoxy bonded to an Al honey-comb core. Silver was chosen as the reflective material. Because Ag does not adhere well to graphite epoxy, an adhesion promoting film of Cu (200Å) was first deposited onto the graphite epoxy facesheet, followed by Ag (1000Å). Two atomic oxygen protective coatings were deposited on top of the Ag: Al_2O_3 (200Å), which is also an adhesion promoting layer, followed by a outer protective coating of SiO_2 (700Å). All films were deposited by electron beam evaporation without breaking vacuum.

The purpose of this study was to evaluate the atomic oxygen durability of these first generation solar concentrator coupons. This paper addresses the effect of atomic oxygen erosion processes on optical performance of SDPM concentrator coupons.

EXPERIMENTAL PROCEDURES

Surface Characterization

The pristine and atomic oxygen exposed coupons ($\approx$3.75 cm^2) were characterized using several photographic techniques. Black and white macrographs ($\approx$1.75X) of the entire coupon were taken using a Polaroid MP.4 Landcamera. Polarized and non-polarized macrographs (6.5-32X) were taken of characteristic defects using a Wild Makroskop M400. Characteristic electron micrographs were taken of both pristine and atomic oxygen exposed coupons (after coating the coupons with $\approx$200Å Au) with a JEOL 840-A scanning electron microscope (SEM).

Reflectance Measurements

Solar reflectance and solar absorptance measurements were obtained using a Perkin-Elmer Lambda-9 UV/VIS/NIR spectrophotometer operated with a 60 mm diameter barium sulfate coated integrating sphere. The specular reflectance was measured at an 8 degree angle from normal incidence, with an aperture solid angle of 0.096 steradian (230 X 320 mrad aperture). Integrated solar reflectances were obtained by measuring the spectral reflectance over the wavelength range of 250-2500 nm. A computer program was used to convolute this spectrum into the air mass zero (AMO) solar spectrum over the same wavelength range[4]. Two measurements of each coupon were made; the coupon was scanned in the center and then rotated 90 degrees and scanned again. Average values of solar hemispherical, solar specular and solar diffuse reflectance were obtained by averaging the results of these two scans. The solar absorptance was calculated by subtracting the solar hemispherical reflectance from 1 because the transmittance was equal to 0.

Atomic Oxygen Exposure

Coupons were exposed to an atomic oxygen environment in a Structure Probe, Inc. Plasma Prep II asher. This asher generates a plasma by exciting ambient air with 100 W of continuous RF power at 13.56 MHz[5]. The operating pressure was 0.5-1.0 x 10^{-2} torr. The plasma is composed of oxygen and nitrogen ions and atoms[6]. The nitrogen species have a negligible effect in the erosion processes[11]. The effective atomic oxygen fluence was calculated based on mass loss data of dehydrated 5 mil Kapton^R H polyimide, which was ashed with the coupons. An erosion yield of 3 x 10^{-24} cm^3/atom for the Kapton^R was assumed based on space flight data[7,12].

RESULTS AND DISCUSSION

Surface Characterization

Macroscopic examination of the pristine concentrator coupons revealed several fabrication and handling defects. The types of defects and their population varied greatly from coupon to coupon. These defects included scratches, macroscopic bumps, dendritic regions, porosity, haziness and pin hole defects. Examples are shown in figure 1. The dendritic regions have branching arms which is characteristic of dendrites (see figure 1c). Figure 1d is a polarized macrograph of figure 1c showing porosity which was often found to be associated with dendritic regions. Upon SEM examination, the regions which appear to be hazy to the unaided eye were found to be microscopic nodules. The chemistry of the nodules or the dendrites is not currently known, but dendrites were removed with the protective and reflective coatings by peeling with tape. This suggests that the dendrites developed during the evaporation of the Cu and/or Ag. Pin hole defects were present on all coupons. Coupons which contained more fabrication and handling defects had lower solar specular reflectances than coupons which had fewer pristine defects.

Atomic Oxygen Exposure

The optical properties of the solar concentrators before and after atomic oxygen exposure are listed in table 1. The change in solar specular reflectance upon ashing to an equivalent of 0.39 years exposure for coupon A was within instrument error and was determined to be negligible. Scanning electron microscopy examination revealed atomic oxygen erosion damage, particularly at scratched regions. Macrographs of coupon B after 0.44 equivalent years of exposure showed evidence of atomic oxygen undercutting at scratched regions and at pinhole defects.

Moderate fluence exposures of coupons A and B resulted in small reflectance changes and limited atomic oxygen damage. Coupons C and D were ashed iteratively to higher fluence levels (1.08 equivalent years of exposure) to document reflectance changes as a function of atomic oxygen fluence. Figure 2 shows macrographs of coupon D: pristine, half way through the atomic oxygen exposure testing (fluence of 1.57 x 10^{21} atoms/cm^2) and after atomic oxygen exposure to a fluence of 3.1 x 10^{21} atoms/cm^2. Figure 3 shows the corresponding specular reflectance spectra. Similar to coupon D, defects in coupon C were found to be susceptible to atomic oxygen attack. Figure 4 shows preferential attack at a porous defect. Half way through the atomic oxygen exposure life, it became apparent that the protective and reflective films had torn at regions which were undercut

extensively (see figure 4b). With continued ashing, the tearing was found to propagate (see figure 4c).

Tearing of the reflective and protective films allows exposure of more substrate material which further erodes, thus continuing the undercutting-tearing propagation process. A circular defect which was undercut and torn can be seen in figure 5. The approximate extent of undercutting at the various ashing intervals is shown by the white rings around the defect areas. If tearing did not occur, it might be expected that the undercut region would reach a diameter limit[6,8].

The undercutting-tearing propagation process was evident and easily documented during iterative ashing. However, it was difficult to determine if the tearing was due to the ashing alone or to changes in the environment during the iterative vacuum and air exposure of the ashing process. If external influences such as moisture and air exposure caused tearing of the reflective and protective coatings, then it is possible that the undercutting-tearing process will not be a problem in LEO. To determine if tearing might occur in space, two pristine coupons (E & F) were ashed continuously to the same effective fluence as coupons which tore when iteratively ashed. Both coupons E and F were found to have torn reflective and protective coatings at extensively undercut regions once they were taken out of the asher. Closer examination of these coupons provided evidence for atomic oxygen undercutting at pin hole defect sites. Figure 6 shows the same region before and after atomic oxygen exposure on coupon E. Notice that several large pin hole defects, indicated as black spots in the pristine polarized macrograph (circled in figure 6a), appear to be in the center of the large atomic oxygen eroded regions of the ashed coupon as shown in the micrograph in figure 6b. Other small dark spots are shown in the micrographs of the pristine and ashed coupon. These spots must be superficial defects on the protective layers since they were not atomic oxygen transparent (i.e. permeable). Examination of coupon F also revealed evidence of preferential atomic oxygen erosion at scratched regions.

To conclusively determine whether the tearing occurred during ashing, two pristine coupons (G & H) were positioned in the asher chamber so that visual examination could occur insitu. During this test it became evident that tearing and subsequent curling of the protective and reflective coatings did occur during ashing. This observation indicates that the undercutting-tearing propagation process could occur in LEO and is a potential threat to solar concentrator durability.

Plots of solar specular reflectance verses fluence for coupons C, D, E and F, show that the rate of decrease in the specular reflectance was slightly greater for coupons which were ashed iteratively (see figure 7). The error bars in this graph indicate the range of reflectance obtained by rotating the coupon 90°. Large variations, particularly in coupon C, are attributed to the aperture window being rectangular. This causes some difference in areas to be scanned on the same coupon. In the case of C, a band of defects were predominantly visible in one reflectance scan, and only partially visible in the 90° scan. The average decrease in solar specular reflectance of the iteratively ashed coupons (C & D) was 0.137, while at the same fluence the average decrease in solar specular reflectance of the continuously ashed coupons (E & F) was 0.070. Similarly, increases in the solar absorptance are greater for the coupons iteratively ashed (an average change of 0.043) than for

coupons continuously ashed (0.029). It appears that iterative ashing caused an acceleration of the undercutting-tearing propagation process and, thus, was found to provide a more severe environment for atomic oxygen simulation than continuous ashing to the same fluence level. Each air exposure after vacuum ashing can allow accelerated tearing of the undercut coatings to occur which allows further atomic oxygen attack in subsequent ashing. Results of the continuously ashed coupons for 1 year equivalent exposure showed a decrease in solar specular reflectance between 0.051 and 0.079, and an increase in solar absorptance between 0.022 and 0.031. Extrapolating these results linearly to 15 years (the expected lifetime of the concentrator) would yield a decrease in solar specular reflectance between 0.764 and complete specular reflectance loss, and an increase in solar absorptance of between 0.333 and 0.472.

Using scanning electron microscopy, the atomic oxygen erosion processes at defect sites were examined in detail. If the original atomic oxygen transparent defect is very small ($< 5\mu m$) it appears that the undercut region reaches a limiting size and tearing of reflective and protective films does not generally occur. Extensively undercut larger defects caused tearing of the unsupported film. Silver oxide was found to be present at many of the torn cracks and defect openings. If the torn region becomes large enough, the protective and reflective layers which are now unsupported, start to curl. This leads to exposure of large regions of the underlying substrate and lets the atomic oxygen react with fresh, previously protected substrate material, as shown in figure 8a. As the undercutting-tearing propagation cycle continues more undercut regions run into each other. Eventually the protective and reflective films not only curl but flake off, as can be seen in figure 8b. Extensive undercutting damage of this kind leads to specular reflectance loss (resulting in decreased system efficiency) and solar absorptance increases (resulting in increased heating and warpage of the solar concentrator) as well as structural damage to the concentrator.

By examining areas where the reflective and protective films have flaked off, the atomic oxygen plasma erosion process was further evaluated. As the atomic oxygen attacks the epoxy, a rounded bottom profile forms (see figure 9a). A very slight angled chamfer at the protective coating interface can be noticed which is attributed to a double dimple effect for wide defect width-to-coating thickness ratios[3]. As erosion continues in large defects, it eventually reaches the first graphite layer. Because the erosion yield of graphite is less than that for epoxy, more of the impact atoms are scattered and the undercutting region is spread out wider, this can be seen in figure 9b. As atomic oxygen attack continues, the graphite fibers are slowly eroded away. Erosion of the graphite fibers can be seen in the center of the undercut regions in figure 9b; these regions are located below the original defect window. A close up of this fiber erosion as shown in figure 9c.

It was found that regions which did not contain fabrication and handling defects did remain protected, unless they were invaded by neighboring defect sites through undercutting and tearing. Thus, atomic oxygen transparent defects were responsible for the degradation of the optical properties. In order to maintain the specular reflectance and solar absorptance of solar concentrators in LEO, the defect concentration must be decreased. In addition, reflectance durability could be improved if the tearing phenomena was better understood and the films ability to resist tearing

increased. Studies have shown that the application of a surface tension leveling coating can reduce the density of atomic oxygen transparent defects by an order of magnitude[9]. A new, fabrication technique for producing solar concentrators for SSF based on replication of ultra smooth surfaces is currently being developed. This technique appears to be very promising for reducing the number of surface defects. Testing of these concentrator coupons is being planned.

CONCLUSIONS

First generation solar concentrator coupons contained fabrication and handling defects which caused low pristine solar specular reflectances. Atomic oxygen erosion was observed to occur at defect sites, particularly at scratches, porous regions and pin hole defects. Extensive undercutting and curling of reflective and protective coatings were found to be promoted through an undercutting-tearing propagation process. Iterative air plasma ashing appeared to cause an acceleration of the undercutting-tearing propagation process and was found to provide a more severe environment than the continuous atomic oxygen exposure that would occur in the LEO environment. The solar specular reflectance of these first generation solar concentrators decreased between 0.051 and 0.079 for 1 year equivalent exposure. Whereas the solar absorptance increased between 0.022 and 0.031. The expected lifetime of the solar concentrator is 15 years. It is concluded that atomic oxygen transparent defects are responsible for degradation of optical properties; therefore, to maintain the specular reflectance and solar absorptance of solar concentrators, the defect concentration must be decreased. In addition the ability of the reflective and protective films to resist tearing when undercut must be improved. Finally, a new fabrication technique for producing solar concentrators for SSF based on replication of ultra smooth surfaces is being considered which may be promising for decreasing the number of atomic oxygen transparent defects.

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Table 1. Effect of Atomic Oxygen on Optical Properties of Solar Concentrators for SSF

Coupon	Test	Exposure Condition	Solar Specular Reflectance			Solar Absorptance			Equivalent Years on SSF+
			Initial	Final	Change	Initial	Final	Change	
A	Ashed (out once) (2 cycles)	1.12 E 21 atoms/cm^2*	0.832	0.840	+0.008	0.151	0.139	-0.012	0.39
B	Ashed (out once) (2 cycles)	1.25 E 21 atoms/cm^2*	0.869	0.838	-0.031	0.117	0.125	+0.008	0.44
C	Ashed Iteratively (6 cycles)	3.10 E 21 atoms/cm^2*	0.836	0.714	-0.122	0.141	0.180	+0.039	1.08
D	Ashed Iteratively (6 cycles)	3.10 E 21 atoms/cm^2*	0.878	0.726	-0.152	0.107	0.154	+0.047	1.08
E	Ashed Continuously (1 cycle)	3.10 E 21 atoms/cm^2*	0.861	0.806	-0.055	0.123	0.147	+0.024	1.08
F	Ashed Continuously (1 cycle)	3.10 E 21 atoms/cm^2*	0.860	0.775	-0.085	0.126	0.160	+0.034	1.08

* Kapton equivalent fluence based on an erosion yield of 3.0×10^{-24} atoms/cm^2
+ Based on an orbit averaged solar facing flux requirement of 9.1×10^{13} atoms/cm^2

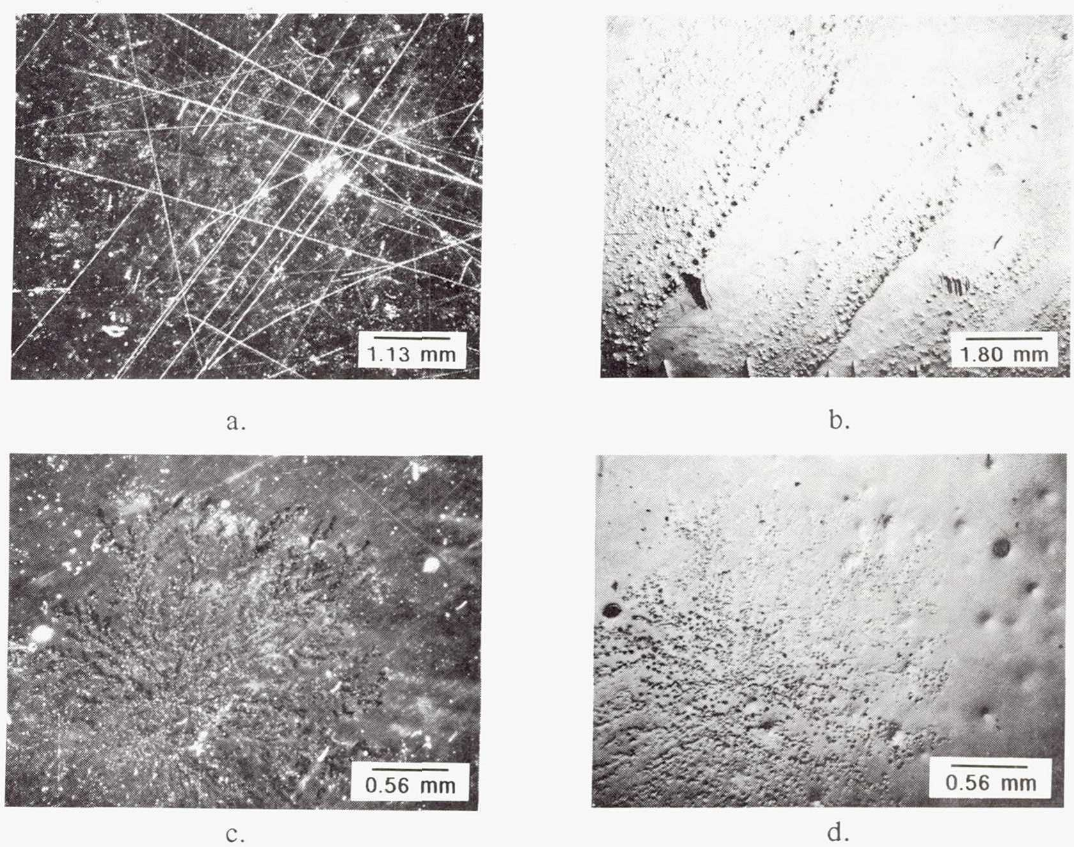
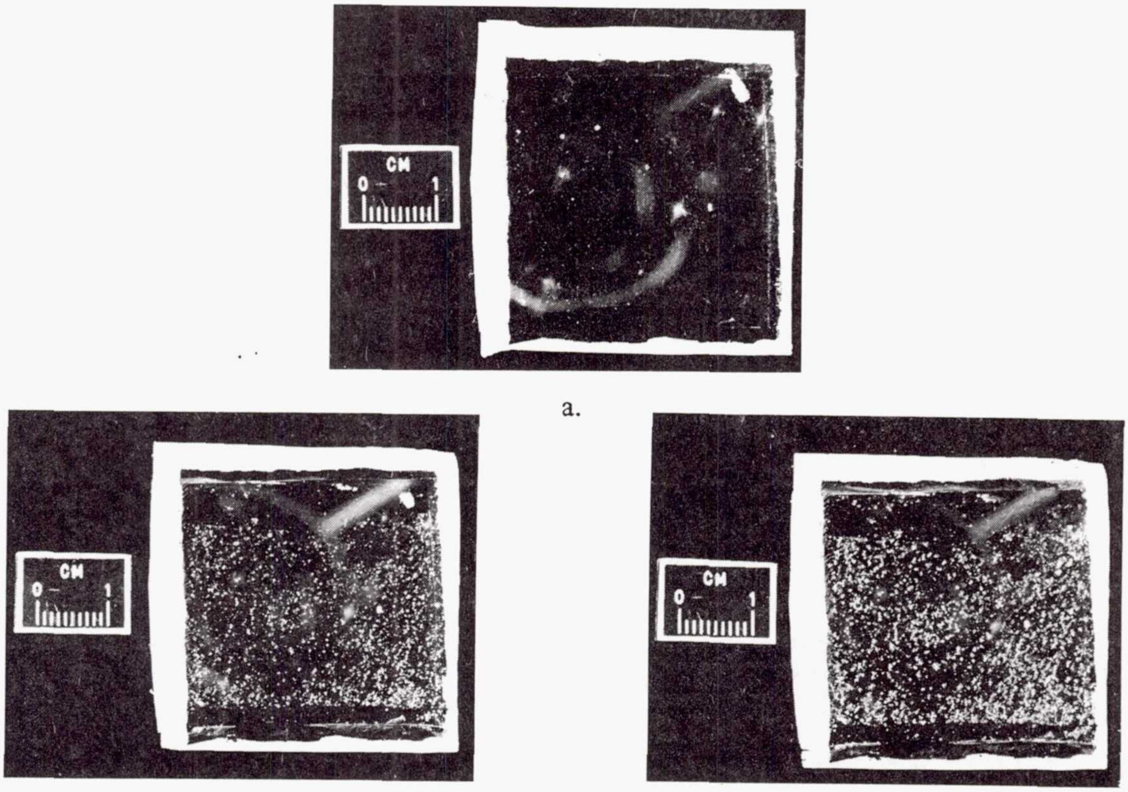


Figure 1. Fabrication and handling defects on as received coupons: a. scratches, b. macroscopic bumps, c. dendritic region, d. polarized micrograph of 1c showing porosity associated with dendritic regions.



a.

b.

c.

Figure 2. Concentrator coupon D: a. pristine, b. fluence = 1.57 x 10^{21} atoms/cm^2, c. fluence = 3.1 x 10^{21}atoms/cm^2.

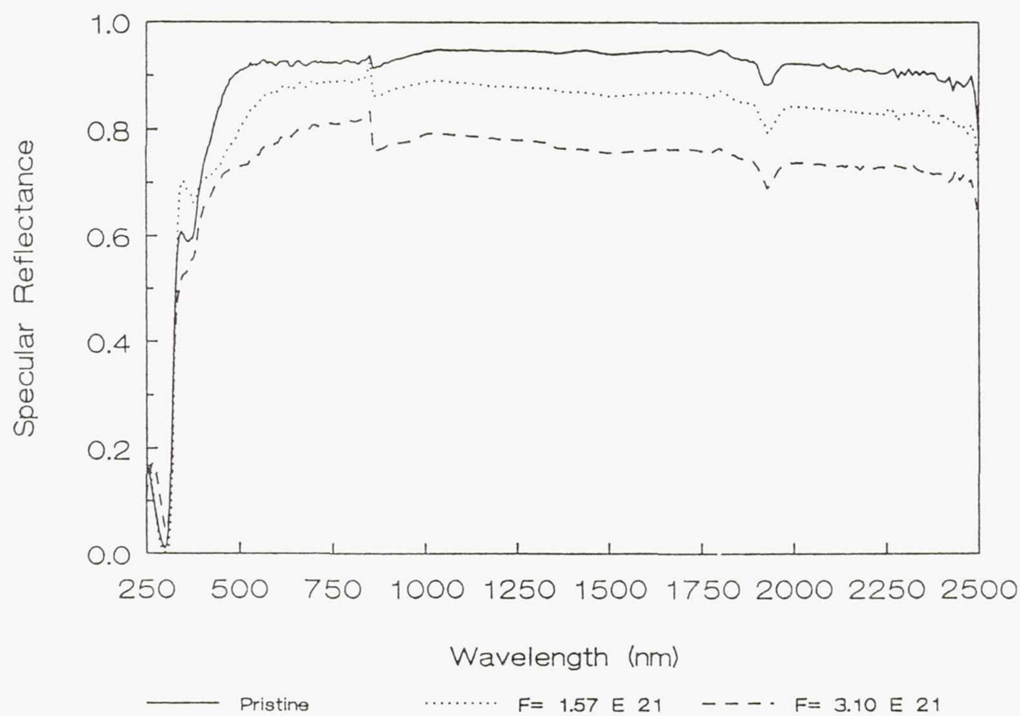
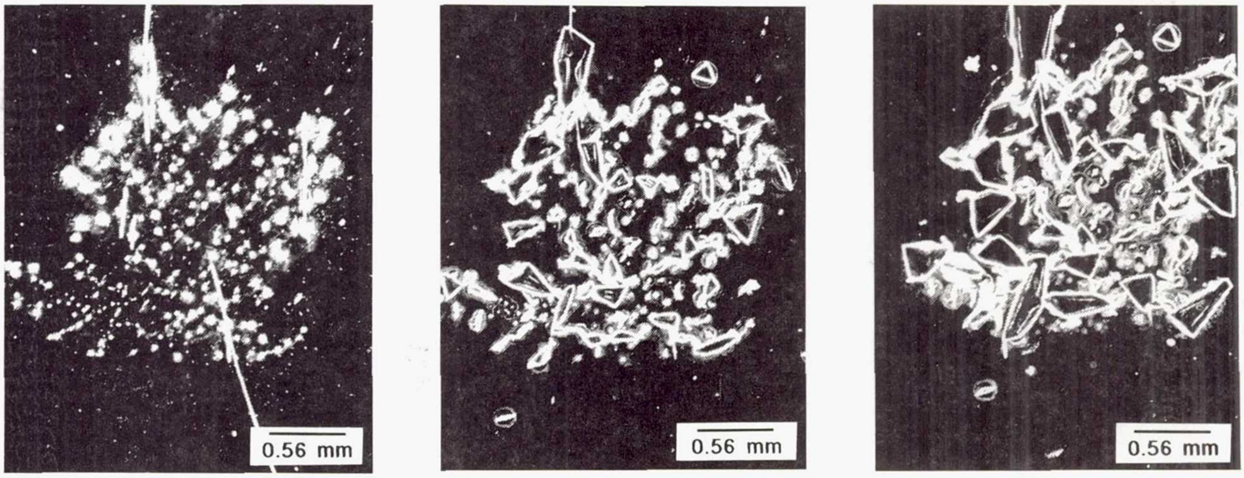
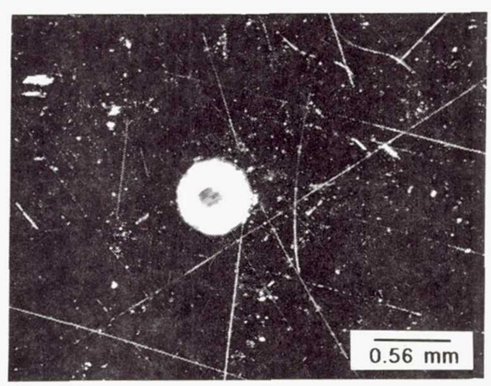


Figure 3. Specular reflectance of coupon D.



a. b. c.

Figure 4. Preferential atomic oxygen attack at a porous region on coupon C: a. Pristine defect, b. reflective and protective films over undercut regions have torn (F= 1.57×10^{21} atoms/cm^2), c. undercutting-tearing process propagates (F= 3.1×10^{21} atoms/cm^2).



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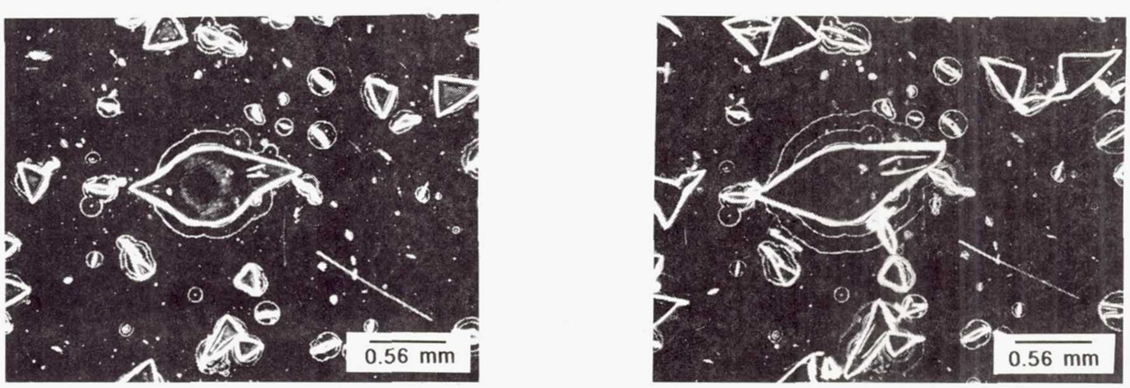
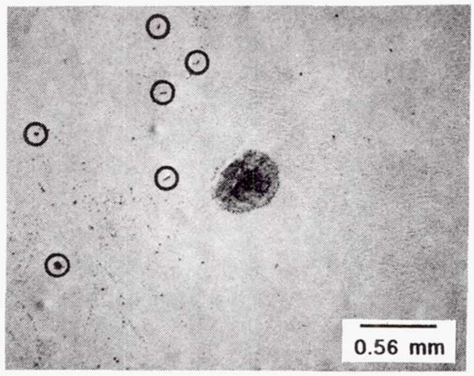


b. c.

Figure 5. Atomic oxygen undercutting and tearing at a circular defect. Concentric white rings indicate the extent of undercutting at various fluence levels: a. pristine, b. F= 1.57×10^{21} atoms/cm^2, c. F= 3.1×10^{21} atoms/cm^2.



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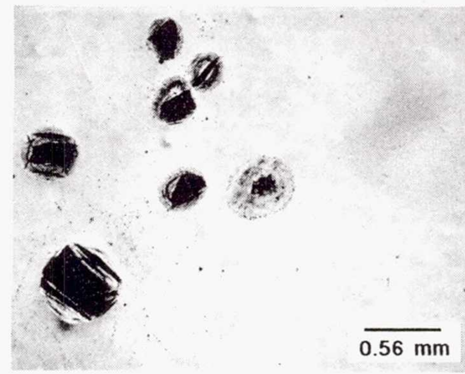


b.

Figure 6. Atomic oxygen undercutting at pin hole defects. Several pin hole defects in the pristine coupon (circled in 6a) are centered in the atomic oxygen eroded regions (6b): a. pristine, b. F= 3.1 x 10^{21} atoms/cm^2.

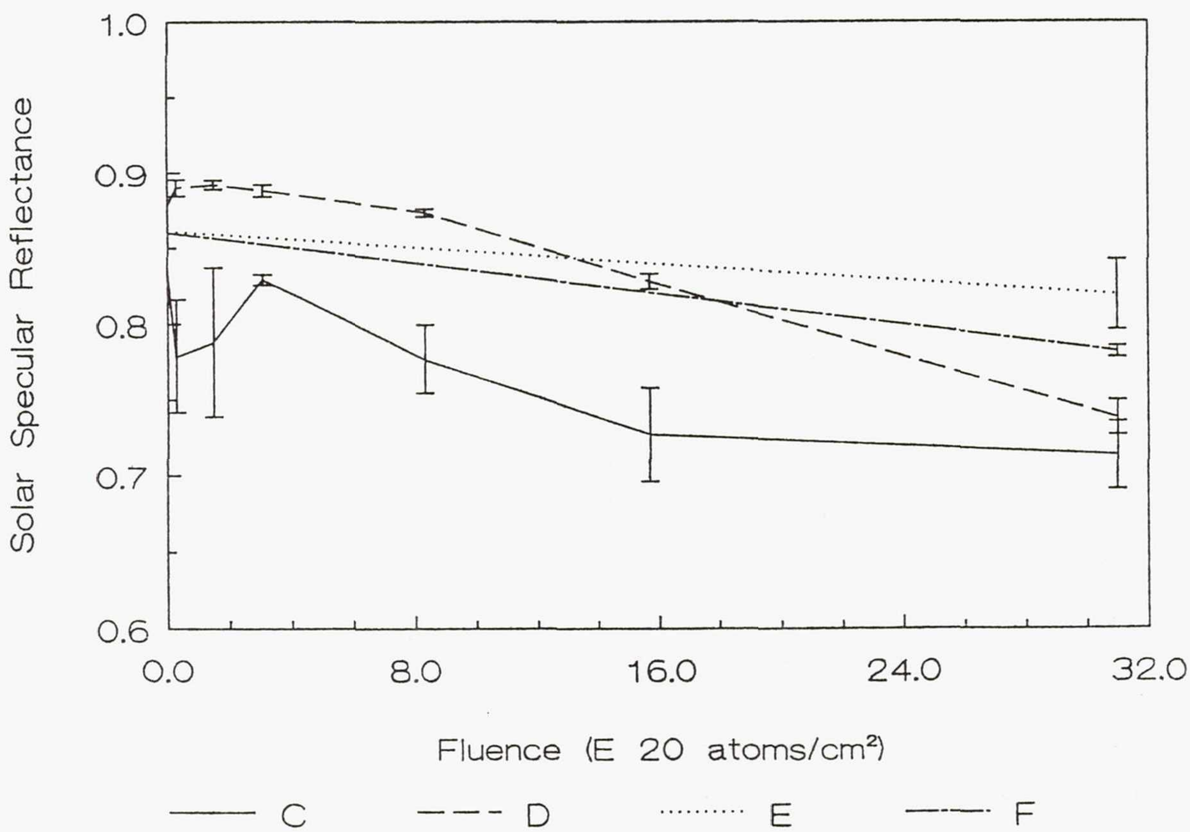
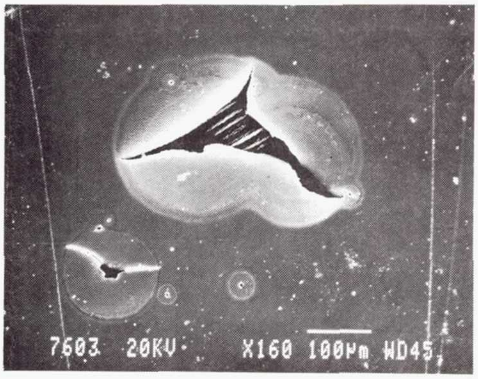


Figure 7. Solar specular reflectance verses atomic oxygen effective fluence for coupons C, D, E and F.



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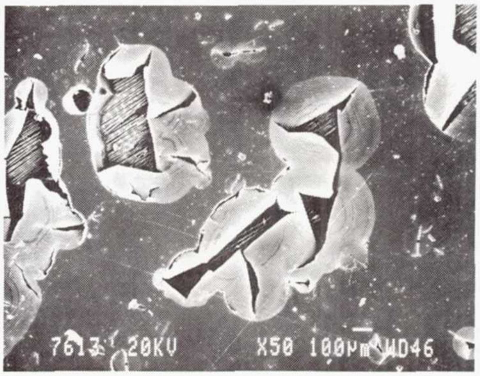
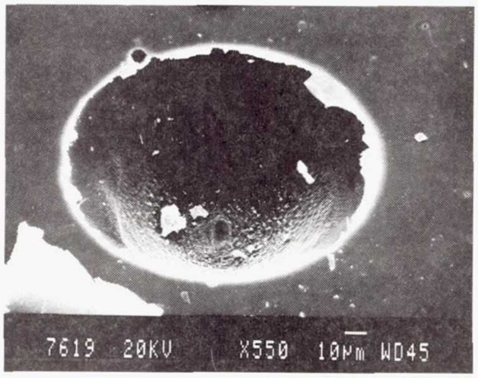


b.

Figure 8. Scanning electron micrographs of undercut regions: a. exposure of graphite fibers due to tearing of the reflective and protective films, b. flaking off of unsupported films at extensively undercut regions.



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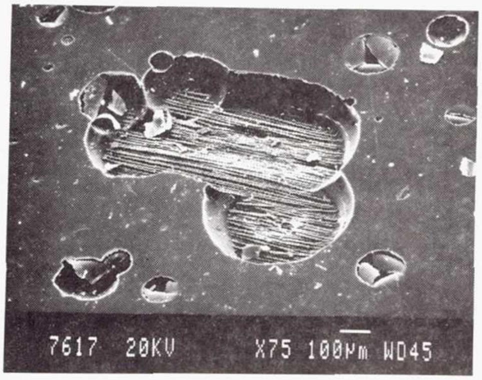

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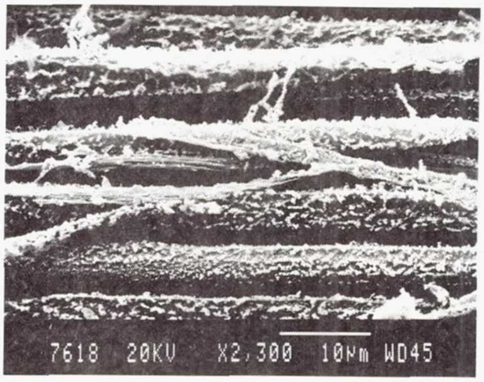

c.

Figure 9. Atomic oxygen erosion processes: a. epoxy erodes with a rounded bottom profile, b. more atomic oxygen atoms scatter at the graphite fiber-epoxy interface (due to the lower erosion yield of graphite) resulting in wider undercut regions, c. graphite fiber erosion below an original defect window in 9b.

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13. ABSTRACT ***(Maximum 200 words)***

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14. SUBJECT TERMS
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